# This Page Is Inserted by IFW Operations and is not a part of the Official Record

## **BEST AVAILABLE IMAGES**

Defective images within this document are accurate representations of the original documents submitted by the applicant.

Defects in the images may include (but are not limited to):

- BLACK BORDERS
- TEXT CUT OFF AT TOP, BOTTOM OR SIDES
- FADED TEXT
- ILLEGIBLE TEXT
- SKEWED/SLANTED IMAGES
- COLORED PHOTOS
- BLACK OR VERY BLACK AND WHITE DARK PHOTOS
- GRAY SCALE DOCUMENTS

### IMAGES ARE BEST AVAILABLE COPY.

As rescanning documents will not correct images, please do not report the images to the Image Problem Mailbox.



- ⊕ No. 813,616
  - 1SSUED May 27, 1969
  - ① CLASS 92-62 C.R. CL.

## © CANADIAN PATENT

MANUFACTURE OF CELLULOS IC. PRODUCT

Leo J. Bernardin, Appleton, Wisconsin, U.S.A.

Granted to Kimberly-Clark Corporation, Neenah, Wisconsin, U.S.A.

(1) A PPLICATION No. 974,750 (12) FILED Nov. 3, 1966

PRIORITY DATE Dec. 13, 1965 (513, 352) U.S.A.

No. OF CLAIMS

This invention relates to crepe paper sheet material and to methods of making the same.

Crepe tissue products have a general utility in applications where absorbency is a factor. These include, for example, household and industrial wipes, towels, packaging, cushioning materials and the like.

The creping of paper on a Yankee drum surface is well known. It involves crowding the tissue paper sheet in the longitudinal direction while it is adhered to the drum to cause disruption of fiber-to-fiber bonds, that is, to limit fiber-to-fiber bonding. The creping action may take place when the sheet is well dried on the Yankee or while it is in the wet, somewhat plastic state, as is known to the art.

10

The crepe tissue sheet is constituted by a plurality of successive hills and valleys extending generally, though not completely uniformly, in a direction transversely of the sheet. This hill and valley effect characterizing crepe tissue webs causes the web material to absorb liquids and the like in such a manner that the absorbed liquids move preferentially transversely in the sheet. That is, the usual crepe tissue product has a high degree of directionality, particularly as to absorbency. In specific application, for example, a drop of fluid deposited in the usual tissue having any significant degree of crepe will be absorbed usually in an elliptical pattern. The long axis of the ellipse will coincide with the valley or transverse direction of the crepe sheet.

A primary object of this invention is to provide absorbent crepe sheet material which is improved as to

uniformity of absorbency. In effect, a more square sheet as to absorbency characteristics for oil, water and the like is achieved.

An important object of the invention is to provide an absorbent crepe paper sheet material having a pronounced nap.

Yet another object of the invention is to provide an absorbent crepe sheet material constituted by a multiplicity of successive hills and valleys wherein the valleys are bridged by fibers which aid wicking of fluids through the sheet.

10

20

Another object of the invention is to provide an improved process for the production of crepe paper wherein, by the creping action, bonding between fibers is not only reduced but a substantial proportion of fibers are caused by the creping action to separate from others of the fibers and to, in effect, pop up from the surface, thereby opening up the pores between fibers and providing a nap surface on a sheet.

These and other allied objects of the invention will be more fully understood by reference to the following detailed description and accompanying drawings wherein:

Fig. 1 is a flow chart illustrating steps in a preferred embodiment of the invention;

Fig. 2 is a fragmentary, greatly enlarged transverse view illustrating the crepe product; and

Fig. 3 is a somewhat idealized view of a crepe shect in accordance with the invention, the fiber orientation being indicated largely is longitudinal for the sake of clear representation.

In accordance with the invention I incorporate in a papermaking furnish with the usual papermaking pulp fibers a substantial quantity of significantly stiff crosslinked wood pulp cellulosic fibers of papermaking length. The

characteristics of the stiff fibers which make them particularly useful in the practice of the present invention include: a substantial lack of interfiber bonding capacity for each other and for the papermaking fibers in the wet and dry state, naturally occurring kinks and curls of the original wood pulp fibers set in the fibers by crosslinking, a substantial fiber rigidity but subject to resilient compression when wet, and a distinct capacity for being wetted by aqueous fluids. The furnish is a uniform blend of the fibers which are interfelted together in a sheet by waterlaying (Fig. 1). The conventional steps of dewatering and wet pressing follow and commonly the sheet is presented on a Yankee dryer surface to a creping blade.

10

.,30

The sheet formation, water content reduction and wet pressing provide a wet sheet in which the interfelted fibers are pressed together and lie substantially parallel to the same plane. The usual cellulosic papermaking fibers, as is well known, have an inherent fiber-to-fiber bonding capacity. This capacity is very pronounced when the sheet is dry to the touch and is commonly termed hydrogen bonding. In the somewhat wet plastic state a significant, though lesser fiber-to-fiber bonding capacity, exists between papermaking fibers. The stiff crosslinked fibers lacking in significant bonding capacity but interfelted with the papermaking fibers become retained by the latter. Additionally, the kink and curl of the crosslinked · fibers and the resilient compressibility of the fibers apparently permits a significant force to be exerted upon the papermaking fibers serving as retainers. Then when the sheet is dried on the Yankee to effect at least some drying, fiter-to-fiber bonis develop between the papermaking fibers. The web is then

presented to the creping blade, and bonds between the cellulosic papermaking fibers are disrupted by creping action. The more dry the sheet as it is presented to the blade the more it adheres to the Yarkee dryer surface and the greater is the disruptive effect of the blade. Additionally, usually the greater the adhesion the more of the hill and valley effect common to crepe sheets is attained.

Release of the bonds between the cellulosic papermaking fibers by creping action is facilitated by the stiffened fibers. These fibers, having been held under compression in the plane sheet, urge the cellulosic retaining fibers apart, providing for very open pores between fibers. More noticeable visually is the development of a nap on the sheet surface. This is occasioned by the stiff fibers springing up from the web surface as they are released. Importantly, these stiff fibers project in all directions within and from the sheet surface including bridging of hills and valleys of the crepe sheet.

10

The resulting product of the procedure as described surprisingly exhibits a much more square absorbency characteristic than does crepe sheets of only the usual cellulosic papermaking fibers. With prior art tissues having a distinct crepe, that is, a ratio 1.1:1 and above, the wicking rate for fluids is much greater along the hills and valleys and the absorbency pattern for a drop of fluid, oil or water, is generally elliptical with the long axis in the valley direction. As the crepe ratio increases the disparity between the valley direction and the cross-valley or longitudinal direction of the sheet increases. In the crepe sheet of this invention the absorption characteristic for a drop of fluid at low crepe ratios is substantially circular and very nearly so even at high crepe ratios. Preferably, the crepe ratio is between about 1.2:1 and

. 4 =

3:1. In some instances a nearly circular absorbency characteristic may be obtained with conventional fibers by drawing out a large proportion of the crepe and calendering but this is at the expense of transverse wicking rate. In the present instance transverse wicking rate is maintained and the circular pattern of absorbency is evidence of the increased total wicking rate of the sheet of this invention in the absorption of fluids.

In a preferred embodiment of the invention stiffened wood pulp fibers were prepared by first forming a mat of bleached southern pine pulp; this mat was then saturated with an aqueous solution of dimethylol urea in which the dimethylol was present to the extent of about 12½ by weight. The solution included also as catalyst ammonium chloride to the extent of about 2% by weight of the dimethylol urea. The pulp mat, after saturation with the solution, was pressed to a 200% wet pickup and air dried. This sheet was then defibered in a hammermill to form a fluff consisting essentially of individual fibers. The fluff was cured at about 130-125°C. in order to effect crosslinking.

Under the circumstances of the example the dimethylol urea solution penetrates the fiber and is primarily within the pulp fibers rather than between fibers as is common in the production of wet strength products. The heating of the impregnated material is effective to set the crosslinking agent within the fibers to materially stiffen the fibers and to change to some extent the surface character of the fibers. The fibers, however, are readily dispersible in water and are suitably washed in water after curing for use in the practice of the invention. Washing removes catalyst and any unreacted soluble agents.

- 30

20

10

A usual papermaking pulp for tissue purposes, that is, a 50-50 blend by weight of bleached softwood sulfite pulp and a spruce kraft pulp were made up as a papermaking furnish. For this purpose the pulp blend was refined to a Canadian Standard Freeness of about 550. Additionally, the pulp combination included 1% by weight based on the pulp weight of a wet strength resin. About 30% by weight of the stiffened fiber in aqueous suspension and produced as described above was added to 70% by weight (air-dry fiber basis) of the pulp blend to form the papermaking furnish. A tissue sheet was formed on the papermaking machine in customary manner, that is, by passing the furnish at about 0.2% consistency to a wire of a Fourdrinier machine. From the Fourdrinier wire the formed sheet moved on a felt and then to a highly polished surface of a Yankee dryer against which it was laid by means of a press roll, the paper sheet exhibiting excellent adherence to the dryer. A suitable mechanical arrangement for the purpose, including creping on a Yankee dryer, is illustrated in United States patent No. 3,014,832. The sheet as it leaves the press roll tends to expand but, apparently, fibers are retained by the water present sc that the fibers remain parallel to a plane.

In the present instance the sheet on the Yankee was subjected to a surface temperature of about 230°F, and the sheet was well dried (3-5% moisture) before creping. The sheet adhered well to the dryer and the crepe was of excellent uniformity. The crepe ratio was about 1.7:1.

Under the action of the creping blade the usual hills and valleys developed in the sheet. Additionally, the stiffened fibers released in part and protruded in all directions from the fiber surface. Under the microscope many extending fibers appear as substantially individual fibers

while other projecting fibers are in small groups, some of which extend longitudinally of the sheet and bridge a plurality of valleys.

As illustrated in the representation of Figs. 2 and 3, the creped sheet 1 is quite open and the extended stiff fibers 2 are apparent. The profusion of the stiff fibers 5 which bridge hills 3 and valleys 4 (Fig. 3) is evident. The stiff fibers are held in position by interlocking contact with the papermaking fibers 6. The stiffened fibers are not visually distinct from the other fibers and many stiffened fibers do not protrude from the sheet thickness but expand the sheet internally by enhancing fiber separation. This contributes to an improved sheet bulk.

10

30

The inclusion of the stiff crosslinked fibers in the sheet appears to shorten the path length for fluid wicking in the longitudinal direction of the sheet. The transverse sheet direction in which the hills and valleys run is slightly improved as to the rate of wicking in many instances, but the change is small compared with the change in the so-called machine or longitudinal direction c: the sheet. The very material increase in rate of wicking in the longitudinal direction gives rise to almost equal wicking rates in both sheet directions at a 50-60% crosslink fiber level. This is governed to some extent by the amount of stiff crosslinked filer included and to some extent, probably, the crepe ratio and fineness of crepe. As crepe ratio increases, that is, the hills and valleys are closer together, transverse wicking rate tends to increase in the absence of the stiff fiber and to be not materially affected by the stiff fiber introduction. The longitudinal rate of wicking increases with crepe ratio but this is slight in comparison with the increased wicking rate in the longitudinal sheet direction occasioned by the introduction of the stiff fiber.

. 7

A repeated series of tests based on the papermaking furnish described in detail above, but with varying percentages of the stiff fiber, clearly shows the influence of these fibers upon absorbency in the longitudinal direction. The wicking rate as measured over a 3-inch length of the product increases sharply with the inclusion of 10% of the crosslinked fibers and any substantial proportion, even 5%, is effective. At a 20% inclusion of stiffened fiber the wicking rate in the machine direction doubled; at 40% the wicking rate was quadrupled and ag 60% the wicking was six times that of a sheet from a furnish of the papermaking fibers having no stiff fiber. In addition, the wicking rate for the sheet as a whole and the total sheet absorbent capacity increased materially. Also, while the above figures are based on water absorption, oil absorption values for both wicking and total absorption increased to the same extent; for example, the capacity of a sheet to retain oil was doubled by the inclusion of 40% of the stiffened fiber, while the wicking rate for oil was quadrupled.

In the range of 50-60% of the stiff fiber the absorbency characteristic is nearly square for most furnishes for both oil and water absorption. While the sheet is more open, aiding wicking, the fiber bridging effect appears to be an important influence in the fluid conduction in the machine direction. The stiff fibers themselves extend rather heterogeneously and are twisted and curled in various directions though probably a majority of the fibers extend in the longitudinal or machine direction and provide for a considerable wicking action. In many instances the bridging fibers are single fibers. In other instances small groups bundled together, and probably including some papermaking fibers, effect the bridging.

20

The sheet feel, despite the presence of stiff fibers, is that of a relatively soft map surface. This web surface

does not become as harsh on wetting and re-drying as do more tightly bonded sheets. Upon re-drying, the sheet retains the protruding stiff fibers.

The product of the invention recovers quickly from wet squeezing or pressing. Void volume of the product under light pressures common to the applications in which these tissue products are employed is not only greater than for the usual 100% wood pulp furnish but, importantly, is greater under light pressure, i.e., 0.4 pounds/square inch. The introduction, for example, of about 25-50% of the stiffened fiber into the plies of a wiping material is effective to lend considerable rigidity to the structure of the sheet and to maintain absorbent capacity even under pressures.

It is common practice to employ creped sheets in multiply form and the characteristics mentioned are applicable to such superposed sheets. In such instances the conjoint rugosities of adjacents do not match and the composite has approximately the bulk of an equal number of single sheets.

The invention has been described particularly in connection with dry creped sheets, that is, sheets having a moisture upon creping of up to about & bone dry basis.

Similar results are attained when the creping is effected wet, that is, up to about 35% moisture content. The absorbent properties are little affected by drying after creping, due, apparently, to the high degree of resiliency of the sheet. The nap in some instances appears to be slightly less than is attained with dry creping. The noted squareness of absorbency is, however, attained, and the slight difference in the concentration of the fibers providing the nap is not apparently significant.

It will be understood that this invention is susceptible to modification in order to adapt to different usages and conditions and, accordingly, it is desired to comprehend such modifications within the invention as may fall within the scope of the appended claims.

### 813616

The embodiments of the invention in which an exclusive property or privilege is claimed are defined as follows:

- 1. The method of producing an absorptive conformable crepe sheet material which comprises:
- a) forming a papermaking furnish consisting essentially of cellulosic papermaking fibers and stiff crosslinked wood pulp cellulosic fibers of papermaking length characterize! by properties including a lack of interfiber bonding capacity for each other and for the papermaking fibers, naturally occurring kinks and curls set in the wood pulp fibers by crosslinking, a substantial rigidity but subject to resilient compression when wet and dry and a capacity for being wetted by aqueous fluids;
- b) interfelting the fibers of the furnish by waterlaying the furnish to form a wet paper sheet;
- c) subjecting the wet paper sheet in succession to a dewatering action and to wet pressing so that the interfelted fibers are pressed together to be substantially parallel to the same plane;
- d) drying said sheet to a moisture content suitable for creping and to produce fiber-to-fiber bonds between contacting cellulose papermaking fibers whereby interfelted stiff crosslinked fibers are retained; and
- e) creping the sheet subsequent to the said drying operation to urge the sheet into a succession of hills and valleys extending transversely of the sheet to disrupt fiber-to-fiber bonds between the cellulosic papermaking fibers to thereby release the stiff fibers in large measure from the pressed and interfelted relation and to cause the stiff fibers to resiliently urge the cellulosic papermaking fibers apart and to protrude in a multiplicity of directions from and to bridge the hills and valleys of the sheet forming a nap surface on the sheet.

### 813616

- 2. The method according to Claim 1 wherein the sheet is substantially completely dried before creping.
- 3. The method according to Claim 1 wherein the sheet is dried to a moisture content of at least about 35% before creping and is subsequently further dried to an air-dry condition.
- 4. The method according to Claim 1 wherein the stiff crosslinked wood pulp fibers are present in the papermaking furnish to the extent of between about 10 to 60% by weight based upon the total weight of the fiber of the furnish.
- 5. An absorbent crepe tissue paper sheet in the form of a porous, bulky fibrous web having a nap surface and characterized by a succession of hills and valleys, said sheet consisting essentially of cellulosic papermaking fibers interfelted with a substantial proportion of stiff crosslinked wood pulp fibers of papermaking length characterized by properties including naturally occurring kinks and curls set in the fibers by crosslinking, a substantial lack of interfiber bonding capacity for each other and for the papermaking fibers when wet and dry, a capacity for being wetted by fluids and a substantial rigidity when wet and dry, said papermaking cellulosic fibers being bonded together at points of contact and retaining the stiff crosslinked fibers in the sheet, said stiff crosslinked fibers projecting from the hills and valleys of the sheet in a multiplicity of directions and some of said stiff crosslinked fibers bridging hills and valleys of the sheet.
- 6. An absorbent crepe tissue product according to Claim 5 wherein the stiff crosslinked wood pulp fibers are present to the extent of between about 10 to 60% of the fiber weight and the cellulosic papermaking fibers are present to the extent of between about 90 to 40% of the fiber weight.

### 

7. An absorbent crepe tissue product according to Claim 6 wherein the 1.tiff crosslinked fiber is present to the extent of between about 50 to 60% of the fiber weight and the wicking rate in the longitudinal direction of the sheet is substantially the same as the wicking rate in the transverse direction of the sheet.

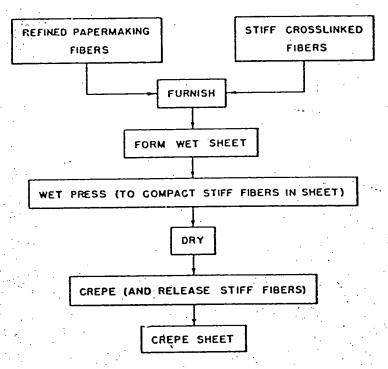


FIG. I

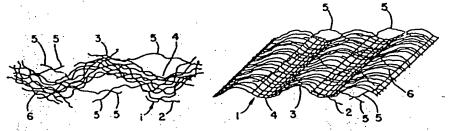


FIG. 2

FIG. 3